SOLVENTLESS COATING EVALUATION AND TEST PROGRAM FOR MARINE USE

OCTOBER, 1981

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IN COOPERATION WITH
AVONDALE SHIPYARDS, INC.

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FOREWORD

This research project was performed under the National Shipbuilding Research Program. The project, as part of this program, is a cooperative cost shared effort between the Maritime Administration and Avondale Shipyards, Inc. The development work was accomplished by Springborn Laboratories, Inc., under subcontract to Avondale Shipyards. The overall objective of the program is improved productivity and, therefore, reduced shipbuilding costs to meet the lower Construction Differential Subsidy rate goals of the Merchant Marine Act of 1970.

The studies have been undertaken with this goal in mind and have followed closely the project outline approved by the Society of Naval Architects and Marine Engineers (SNAME) Ship Production Committee.

Mr. Leon Levine served as Project Manager and Senior Research Scientist. Mr. Charles Parker and Mr. Bernard Baum served as Research Scientists.

On behalf of Avondale Shipyards, Inc., Mr. John Peart was the R & D Program Manager responsible for technical direction of publication of the final report. Program definition and guidance was provided by the members of the 023-1 Surface Preparation Coatings Committee of SNAME, Mr. C. J. Starkenburg, Avondale Shipyards, Inc., Chairman.

Also we wish to acknowledge the support of Mr. Jack Garvey and Mr. Robert Schaffran, of the Maritime Administration. Special thanks are given to the numerous suppliers listed below for their valuable contribution of information.

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- K-T-A Tator Associates, Coraopolis, PA
- U.S. Steel Corporation, Fairless Hills, PA and Boston, MA

EXECUTIVE SUMMARY

With the advent of Clean Air Regulations, the Marine Industry has been confronted with reducing the amount of volatile organic compounds liberated during ship painting operations. One means of eliminating or sharply reducing effluent solvent air-pollution is through the use of 100% solids (solventless) coatings. This project is a first step approach to establishing the suitability of using high solids paints and coatings for marine applications.

A survey of more than 50 manufacturers of marine paints was conducted which showed that only a very few produced true solventless coatings --- most of these were epoxy based. Following the survey, several representative solventless materials were selected for screening tests. For this study, the term "solventless" coatings pertained only to 100% solid types and did not include any waterborne coatings. Test control coatings were selected which consisted of standard marine coatings with varying amounts of volatile organic solvents. Both the candidate coatings and the control coatings were similarly applied and subjected to testing representative of marine exposures. These tests included:

- o Boiling water resistance - 500 hours
- o Diesel Oil (#l Kerosene) Immersion at 100°F - 500 hours
- o Salt Spray (ASTM B117-73) - 500 hours
- o Accelerated Weathering (Atlas Weatherometer) - 1000 hours
- o Pressure Immersion (40 psi) - 6 weeks
- o Adhesion (Non-performance test) Dry Elcometer Adhesion Tester

Results of the testing show that the performance can be divided cleanly into three groups:

Group I - the highest in performance, includes the three controls (epoxy-amine adduct, epoxy/ketimine and epoxy coal-tar) and the epoxy-multicomponent coal-tar 100% solids.

Group II - is somewhat lower, primarily due to failure in one or more of the performance tests, for example, boiling water. All coatings in this group are 100% solids. Included are the polyurethane, the epoxy-polyamide, the other coal-tar epoxy, and the epoxy-amine adduct. Best of this group was the polyurethane.

Group III - is the lowest in performance due to some failure in all tests except diesel fuel resistance and pressure. Included here are both 100% solids epoxy-amine formulations and the polyester.

Based on the results of this study, two of the candidate solventless coatings (multicomponent coal-tar epoxy and polyurethane) should be subjected to a controlled ship application under actual shipyard application conditions. Both the polyurethane and coal-tar epoxy demonstrated potential as a underwater bottom anticorrosive coat and the coal-tar could possibly be used as a tank coating in selected areas.

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I. CONCLUSIONS

1.1 Project Results

Hot rolled steel panels, 0.125″ thick and sandblasted to gray metal, 1.5 mil profile, were coated by high pressure air-less spray and cured at ambient conditions (i.e. air-dried). Coated panels were subjected to boiling water (500 hours); diesel fuel immersion at 100°F (500 hours); salt spray resistance by ASTM B-117 (500 hours); accelerated weathering - 1000 hours; pressure immersion in water at 40 psi (six weeks). In addition, adhesion measurements were made using an Elcometer adhesion tester. Eleven individual coatings were applied for test.

The table which follows shows the resin type in each coating, the solids by volume and the color. The numerical ratings are based on the general appearance of a tested panel of each coating type for each test versus a coated, untested panel of each coating type.

For detail on individual test results, see the test tables in Section 2 (Tables III-VII).

From the results in the summary table, it was considered that performance could be divided into three groups:

Group I (best) - The three controls (B,C and E in Summary Table) plus the 100% solids epoxy-multicomponent coating (D).

Group II - The polyurethane 100% solids (A), the 100% solids epoxy-polyamide (L), the lOO% solids epoxy-amine adduct (H), and the 100% solids epoxy-coal tar amine (G).

Group III (Poorest) - The 100% solids white epoxy-amine (J), the 100% solids red epoxy-amine (K), and the polyester (F).

As a general conclusion, it appears from this study that promising results can be expected from some 100% volume solids epoxy-coal tar compositions and possibly 100% solume solids polyurethane compositions. High pressure, air-less srpay equipment does an excellent application job for these 100% solids products. For further detail on coating compositions and manufacturers, refer to Section 2, Table I.

TABLE A

Summary Ratings[1] of Environmental Test Results versus Orginal Untested Panels

TEST	COATING DESIGNATION										
	A	B	C	D	E	F	G	H	J	K	L
Diesel Immersion	10	10	10	10	10	10	10	10	10	10	10
Pressure Test	10	10	10	10	10	10	10	10	10	10	10
Boiling Water	1	8	8	8	8+	8	1	5	1	1	9
Salt Spray[2]	9+	8	5+	8+	8+	5	7	6+	5	2	3
Accelerated Weathering	8	8	9	7	8	1	8	5	8	3	6
Adhesion[3]	9.5	4.0	8.8	6.0	7.0	3.8	10+	9.0	9.0	5.8	8 .0

Resin Type Identification:

A. Polyurethane - 100% solids by volume - color: black

B. Control - Epoxy, amine adduct cured ;42% solids by volume - color: grey

C. Control - epoxy, ketimine- cured;96% solids by volume - color: white

D. Epoxy, multicomponent, coal tar - 100% solids by volume - color: black

E. Control - epoxy, amine cured ;79% solids by volume - color: black

F. Polyester, peroxide cured ; 100% solids by volume - color: grey

G. Epoxy - coal tar amine cured;100% solids by volume - color: black

H. Epoxy, amine-adduct cured ; 100% solids by volume - color: yellow

J. Epoxy, amine cured ; 100% solids by volume - color: white

K. Epoxy, amine cured ; 100% solids by volume - color: red

L. Epoxy , polyamide cured ; 100% solids by volume - color: blue

Footnotes:

(1) Rating scale is 10 (excellent or relatively unaffected) to 0 (very poor or grossly affected

(2) By ASTM B-117 method

(3) Value shown is the actual psi value divided by 100. Not an environmental test.

1.2 Cost Data

The raw material cost on a cents per mil per square foot basis for the coatings tested varied from 0.84 to 1.89. No raw material cost data on the polyurethane coating (A) was available from the manufacturer. The <u>applied</u> cost was estimated by him at about 10 cents per mil per square foot.

To obtain the raw material cost on this basis, there are found to be 0.144 cu. in. (144 sq. in. x 0.001) in a square foot at one mil thickness. There are then found to be 0.00063 gallons in 0.144 cu. in. (0.144 ÷ 231 cu. in. in one gallon).

The formula for calculation of raw material cost is then:

$$\frac{0.00063 \times \text{price per gallon in cents}}{\text{volume solids (in hudredths)}}$$

Examples:

(1) $$\frac{0.00063 \text{ X } 2400}{1.00 \text{ (100\% solids)}} \quad 1.51 \text{ cents}$$

(2) $$\frac{0.0063 \text{ X } 1320}{0.42 \text{ (42\% solids)}} = 1.78 \text{ cents}$$

From these figures, raw material costs are as follows:

	Cents per mil/sq. ft.
B. Control, epoxy amine - adduct, 42% solids, grey	1.78
C. Control, epoxy-ketimine, 96% solids, white	1.42
D. Epoxy, multi-component, coal tar, 100% solids, black	1.18
E. Control, epoxy amine, 79% solids, black	0.84
F. Polyester, 100% solids, grey	1.59
G. Epoxy, amine, coal tar, 100% solids, black	1.01
J. Epoxy, amine, 100% solids, white	1.89
K. Epoxy, amine, 100% solids, red	1.86
L. Epoxy, polyamide, 100% solids, blue	1.66

1.3 Possible Research and Development

Since not too many coating suppliers were prepared to offer for sale true 100% solids sprayable marine-type coatings, it would seem that the issuance of a tentative specification for coatings of the desired quality should be considered.

Only two basic resin types, polyurethane and amine or amide cured polyepoxy, appear to yield satisfactory coating general properties. Search for other types, e.g. acrylic or modified acrylic, should be continued.

In the urethane field, oligomeric urethane resin with pendant functional groups (e.g. carboxyl, hydroxyl and oxazolidine) could yield useful resins with, for example, triisocyanate crosslinker(s). Some tri-functional cyclo adducts (MDI plus carbodiiamide linkage) are available commercially. A typical example of such a material would be Upjohn’s Isonate 143L, useful in solventless two-component systems.

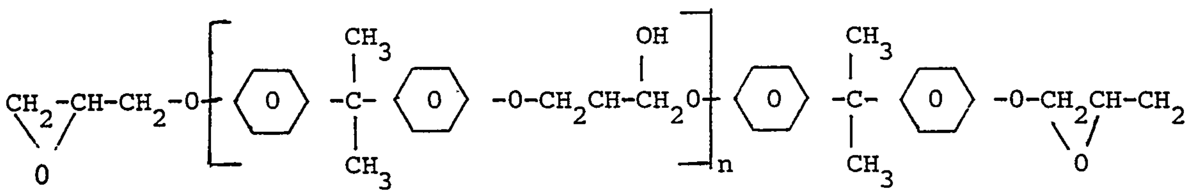
In the current study, coal tar-epoxy compositions had good ratings in all tested properties. Further improvement in the formulation and performance of such resin coating components could yield 100% solids coatings, except for the black color limitation, with quite generally satisfactory application and performance qualities at reasonable raw material prices.

SECTION 2
Project Plan of Action

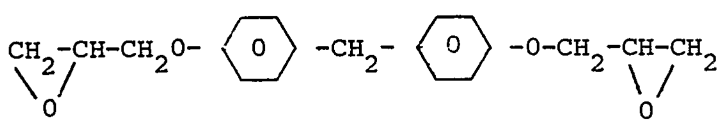
2. PROJECT PLAN OF ACTION

2.1 Background Technical Information

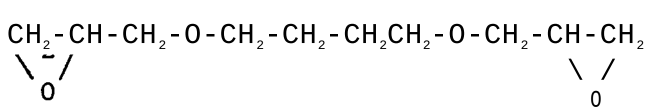
At this time, almost all of the 100% solids paints for marine use which have been tried successfully on a commercial scale are made from epoxy resins. Epoxy resins, chemically, are based on epoxidized bisphenol A or bisphenol F and the use of reactive, epoxidized, viscosity modifiers based on butanediol, or neopentyl glycol. Structural formulas are listed as follows:



Bisphenol A Epoxy Resin



Bisphenol F Epoxy Resin

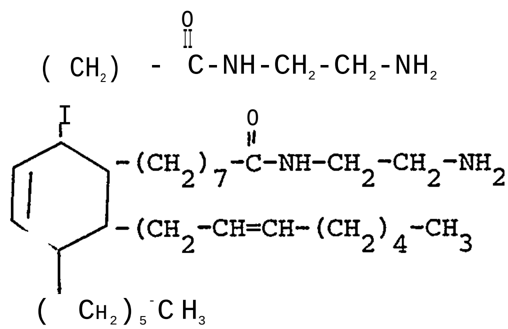


Butanedioldiglycidyl ether

Resins are cured generally at ambient temperatures by polyfunctional amines, polyamides, ketimines or amine adducts whose structures are illustrated below.

$$H_2N-CH_2-CH_2-NH-CH_2-CH_2-NH_2$$

Diethylenetriamine



Polyamide

$$R_2C=N-CH_2CH_2-NH-CH_2-CH_2-N=CR_2$$

ketone blocked polyamine (Ketimine)

$$H_2N-R-NHCH_2CH-R^1$$
OH

Amine adduct

A polyamide type curing agent is formed by reacting a dimer acid with a polyfunctional amine. Illustrated is the reaction product of linoleic acid dimer with ethylene diamine.

The ketimine adduct is the reaction product between a volatile ketone and an amine. After the activated epoxy resin is applied, moisture reacts with the double bond regenerating the amine and causing cure.

An amine adduct type curing agent is formed when an amine is allowed to react with an epoxy. These adducts have lower volatility due to higher molecular weight and have greater reactivity due to the presence of hydroxyls.

When cured, epoxy resins are among the most durable of coatings, (except for gloss loss) and adhesion to metallic substrates is excellent. Cured resins are resistant to water, gasoline and oil which make them ideal coatings for marine use. Such coatings offer excellent corrosion resistance on steel.

Other types of 100% solids paints available are based on either polyurethane or polyester resin technology. Polyurethane paints cure by the reaction of isocyanate with a polyol in the presence of catalyst and moisture.

$$\text{O=C=N-R-N=C=O} + \text{OH-R'-OH} \xrightarrow{\text{catalyst}} \text{-OC-HN-R-NH-COOR}' - \text{O} -_n$$

Certain oligomeric urethane resins with pendant carboxyl, hydroxyl, and oxazolidine functionality, and with the help of triisocyanate crosslinker(s), yield some useful resins for high-solids formation. Pigmented coatings based on these compounds permit the use of conventional spray technology and can be applied at sufficiently high solids to significantly reduce solvent content. There are indications that this technology could be refined to develop solventless urethane coatings of high performance.

Aliphatic polyisocyanates are of interest for this project because of the UV light stability of the urethane coatings formed from them. A number of newer aliphatic diisocyanates have been introduced into the U.S. market. Foremost among these are 4,4'-dicyclohexyl-methane diisococyanate and isophorone diisocyanate.

Trifunctional cycloadducts made of MDI[1] by introducing carbodiimide linkage in the structure are today available commercially (typical example: Isonate 143L, made by Upjohn). Upon heating, an isocyanate group is generated which can be utilized for permanent crosslinking. The carbodiimide linkage can have a dual function: it can be used for further crosslinking, or it can stabilize the polyurethane against hydrolytic degradation. These cycloadducts have been used successfully in solventless, two-component polyurethane systems; however, some of the systems need heat activation.

Polyurethane paints are said to offer the same resistance to water, oil and gasoline as epoxies. Some of the styrene found in polyester paints is volatile and therefore presents an odor problem in use. Besides this problem the solvent and water resistance are not as good as epoxy or polyurethane paints.

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(1) methylenediphenyl diisocyanate

2.2 Objective

The primary objective was to find a marine coating which could be applied at 100% solids by spraying, cured at ambient temperatures (60-80°F) and giving performance qualities in environmental testing essentially equal to those of coatings now in use at shipyards. The control coatings contain volatile organic solvents; it is essential that replacements contain no such materials (e.g. 100% solids) to satisfy air pollution requirements. The only coating method considered likely to yield satisfactory films was high-pressure air-less spray.

2.3 Plan of Action

2.3.1 Selection of Paints

After consultations with the project monitor, the paints listed in Table 1 were selected for applying by air-less spraying. The table also lists the paint characteristics. It is seen that nine out of the eleven paints selected are epoxies. The three control epoxy formulations are solvent based and represent two different types of curing agents and a coal tar base epoxy. The two other paint types used in tests are a polyurethane and a polyester. Paint characteristics and price information are also listed in Table 1.

2.3.2 Sand Blasting of Panels

Hot-rolled steel, approximately 1/8″ thick, was purchased from the mill with mill-scale intact. Panels were cut by the mill to 2″ x 6″, 4″ x 8″ and 6″ x 12″ sizes.

In order to simulate the treatment which the steel receives when ships are painted, panels were sandblasted to a 1.5 mm profile. The height of the profile was measured using a Keane-Tator surface profile comparator. This unit consists of a disc divided into five sections each with a different pattern depth ranging from 0.5 to 4 mils. The comparator disc is placed on the sand blasted panel and the depth profile of the panel is visually compared to that of the standard disc. Panels were sand blasted to a depth which fell between 1 and 2 mm on the comparator. The comparison is made with the aid of a 5x illuminated magnifier.

TABLE I

Characteristics of Paints Used in Study

Panel Designation	Paint Designation and Manufacturer	Resin Type	Color	Price/gal	Comments
Z	Zebron Xenex Corp.,Houston TX	polyurethane	black	(a)	
0	5673 Napko Corp.,Houston,TX	epoxy,42% solids amine adduct	grey	13.20	control
1	5863 ibid	epoxy,96% solids ketimine	white	21.60	control
2	772 CPS Baton Rouge,LA	epoxy,100% multi-component	black	18.80	coal tar
3	5638 Napko	epoxy,79% solids amine	black	10.60	coal tar control
4	3115 General Polymer Cincinnati, OH	polyester, 100% MEK peroxide	grey	25.63	
5	Navitar Jotun Baltimore Baltimore, MD	epoxy,100% amine	black	16.00	coal tar
6	Colturiet TCP/CSF Sigma Coatings Harvey, LA	epoxy, 100% amine-adduct	yellow	18.46	
7	Naviguard Jotun Baltimore	epoxy,100% amine	white	30.00	
9	Decorez 3510 General Polymer Corp.	epoxy, 100% amine	red	29.58	
10	5684 Napko Corp.	epoxy,100% polyamide	blue	26.40	

(a) Not priced per gallon. Sold on a square foot coverage basis.

Sand blasted panels were wrapped in absorbent paper and kept in a constant temperature-humidity room, 70°F, 50% R.H. Additionally, in order to keep moisture low, the wrapped panels were stored in plastic bags containing Drierite, a desiccant.

2.3.3 Mixing of Coating. Materials

2.3.3.1 Coating #Z, Table 1

Mixing information not available. This coating was mixed and applied by its manufacturer. No information supplied.

2.3.3.2 Coating #0, Table 1

Mixed 5 parts of epoxy component to 1 part of convertor by volume. Stirred by propeller-type mixer at slow speed. Mixture yields 41.6% solids by volume. Pot life is 24 hours plus.

2.3.3.3 Coating #1, Table 1

Mixed 4 parts of epoxy component to 1 part of convertor, by volume. Convertor stirred into epoxy by propeller-type mixer at slow speed. Mixture yields 96.4% solids by volume. Pot life is about 4 hours.

2.3.3.4 Coating #2, Table 1

Mixed 4 parts of epoxy component to 1 part of convertor, by volume. Added convertor to base with stirring by propeller-type mixer at medium speed. Mixture yields 100% solids by volume. Pot life is 4 hours plus.

2.3.3.5 Coating #3, Table l

Mixed 1 part epoxy component to 1 part amine convertor, by volume. Added convertor to base with stirring by propeller-type mixer at slow speed. Mixture yields 78.9% solids by volume. Pot life is over 6 hours.

2.3.3.6 Coating #4, Table 1

Mixed 1 gallon base to 1.25 liquid ounces of catalyst. Added catalyst to base while stirring by propeller-type mixer at medium speed. Mixture yields 100% solids by volume. Pot life is 2 hours.

2.3.3.7 Coating #5, Table 1

Mixed 3 parts of base (epoxy) component to 1 part convertor, by volume. Convertor added to base while stirring with propeller-type mixer at slow speed. Mixture yields 100% solids by volume. Pot life is 5 to 6 hours.

2.3.3.8 Coating #6, Table 1

Mixed 4 parts of epoxy (base) component to 1 part convertor by volume. Convertor added to base while stirring with propeller-type mixer at slow speed. Mixture yields 100% solids by volume. Pot life is over 4 hours.

2.3.3.9 Coating #7, Table 1

Mixed 3 parts of base (epoxy) component to 1 part convertor by volume. Added convertor to base while stirring with propeller-type mixer at slow speed. Mixture yields 100% solids by volume. Pot life is over 5 hours.

2.3.3.10 Coating #9, Table 1

Mixed 2 parts base to 1 part hardener, by volume. Added convertor to base while stirring with propeller-type mixer at slow speed. Mixture yields 100% solids by volume. Pot life is 2 hours plus.

2.3.3.11 Coating #lO, Table 1

Mixed 2 parts base (epoxy) to 1 part convertor by volume. Added convertor to base while stirring with propeller-type mixer at slow speed. Mixture yields 100% solids by volume. Pot life - - under 1 hour.

Coatings with mixed pot life of 2 hours or less probably should not be attempted on a pre-mix basis. Such materials are better suited to two-component spray equipment. Actually all coatings except #10 were sprayable, using air-less spray, under laboratory mixing, spraying and clean-out conditions but only those with 4 hours or more of pot life after mixing should be attempted on large Scale operations without benefit of two-component equipment.

2.3.4 Painting of Panels

Panels were coated using Graco air-less spray equipment. At Springborn Laboratories, Inc., a single stage 30:1 Bulldog model was used. Some of the paints could not be sprayed at Springborn Laboratories and were sprayed at Graco Incorporated, Franklin Park, Illinois. At Graco either 25:1 Bulldog Hydrocat plural component pump or a 45:1 King single component pump were used. The ratio given before the pump is the fluid to air pressure ratio and means that for every pound of air going into the pump the fluid can build up the given pressure. Thus at 40 pounds inbound air at a 25:1 ratio the fluid will exit with a theoretical pressure of 1,000 psi. The plural component pump has a feed ratio of 3:1. Detail on equipment used is in Table II.

Wet film thickness was measured using Nordson wet film gages in the 0-20 mil or 4-60 mil ranges.

After prescribed cure time, panels were backed by brushing on a coat of Rust-Oleum #7773 metal primer and air-drying at 70°F and 50% R.H. for at least 24 hours.

During the painting of the panels with the 00% solid equipment, clogging of the spray head was observed and in many cases an in-line, spray head filter had to be used. In some cases the lines were heated in order to get the correct spray pattern. These problems are caused by poor pigment dispersion and high viscosity. A manufacturer simply can not assume that pigment dispersion would be the same for a 100% solid system as it would be for a solvent-containing paint. The problem of viscosity and the necessity of using special equipment is attacked by reformulating so that a paint may be sprayed using conventional plural spray equipment.

The rationale for using a single component airless spray unit was that given a sufficient working time the panels could be easily sprayed. The time needed from start to finish of a spraying operation was about one hour. This included mixing of the paint, test spraying of the pattern, painting the panels and cleaning of the spray equipment. The latter was very laborious. For instance, paint which has a reported pot life of only 0.5 hours would not leave enough time to be applied by a single stage sprayer.

The polyester coating (#4) gave curing difficulty. The manufactuer's literature states the following pot life information:

Temp. %F	Catalyst 1.5 oz/gal Pot Life, min.	1 oz/gal Pot Life, min
60-64	95	180
75	75	19-105
90	32	65

Proportions used were 1 oz/gal and it was found that the panels did not cure overnight. After consulting the manufacturer, the panels were cured at 60°C for 2-1/2 hours.

Pertinent painting information on all coating application is presented in Table II. Six 2" x 6", six 4" x 8", and two 6" x 12" panels for each coating were sprayed. Another panel was included to measure film thickness because the gauge marks remain after use on the panels.

TABLE II

PAINTING OF PANELS

Panel Designation	Sprayed at	Tip	Pressure Inlet psi	Pressure Outlet Base/Catalyst	Temperature of Base/Catalyst	Wet Film Thickness, mils
Z	X[b.]	0.019-0.037	100-200	2800-4200	R	60-70
0	S[c.]	619	32	800	R	16
1	S	515	50	1400	R	10-12
2	S	619	90	2600	R	24-28
3	S	619	50	1400	R	12-14
4	S	619	50	1450	R	12-14
5	G[d.]	523	80	1800/1900	95/100	15
6	G	631	40	2100	R	18
7	G	523	80	1800/1900	95/100	15
9	S,G	525	65	3100	R	21
10	S,G	525	65	3100 (a)	R (a)	21

a. When Hydrocat was used
b. Sprayed at Xenex Corporation (X)
c. Springborn Laboratories, Inc.(S)
d. Graco, Incorporated (G)
R. Room ambient temp, ca.70°F

The photograph below illustrates the results on coatings 2, 3 and 5. All of these are the same type (coal tar-epoxy). Coating #3 is the control for this type. The bottom row shows the untested panels for each coating. The lower half of the tested panels represents the immersed portion. Coating #2 shows severe gloss loss (from chalking) on the immersed portion. Coating #3 shows gloss loss also from chalking plus small areas of blisters at the immersion line (center line in photo). Coating #5 shows slight whitening plus an area of peeling at center of top half.

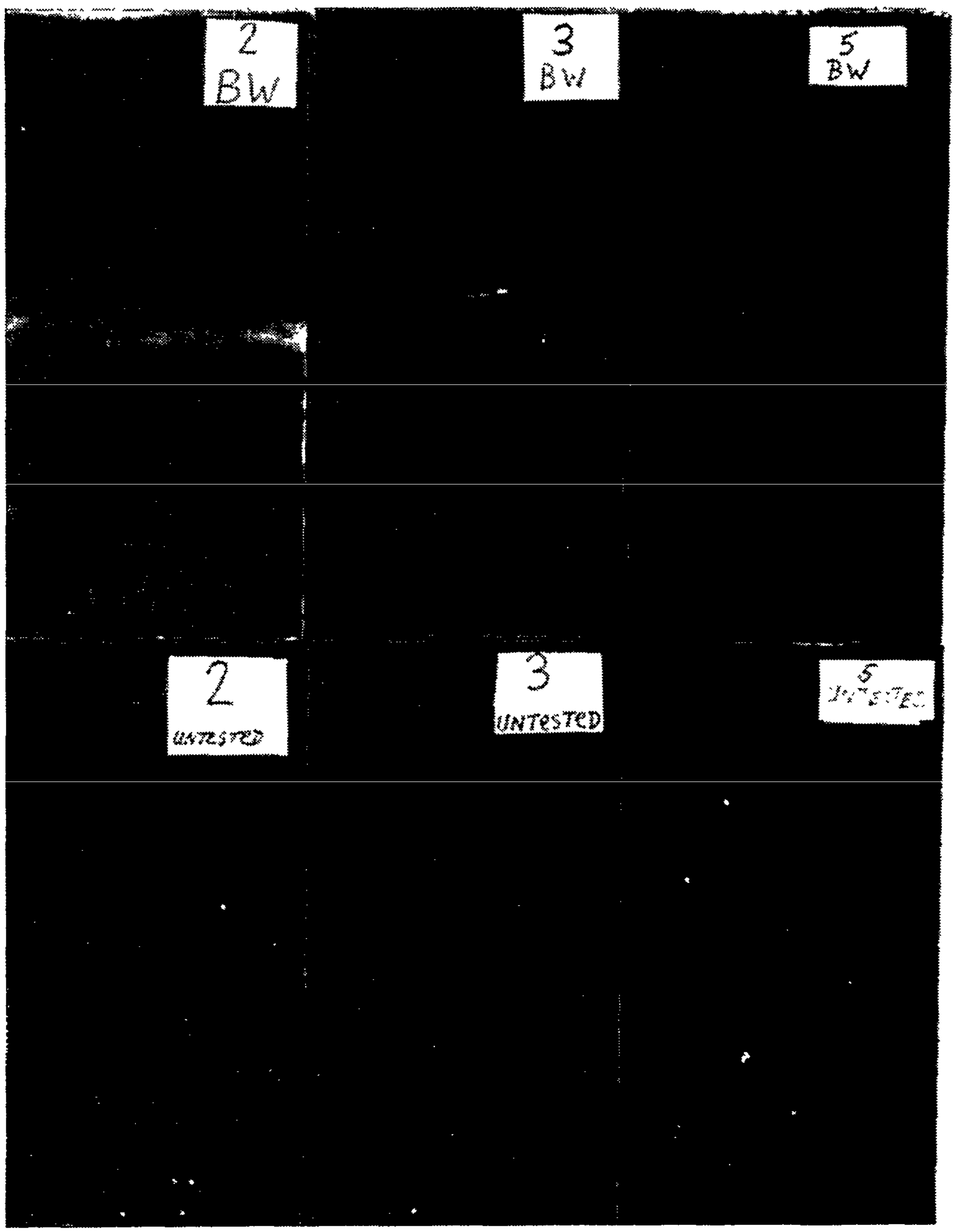


Figure 2.4.1 Boiling Water Test

TABLE III

RESULTS OF BOILING WATER TESTS-500 HOURS

Panel Designation	ASTMD 714-56 Above	Below	% Average Gloss New Panels	% Average Gloss Above	Below	Comment
Z	failed after 8 days		77	-	6	
0	10	10	matte finish			discolored
1	10	10	44	17	37	discolored
2	10	10	79	7	5	
3	10	(a)	37	5 or less	8	top center discolored where water dripped
4	1 sm.cluster 6/med.dense	10	matte finish	-		
5	peeled after 72 hours 6/medium	4/dense	86	13	7	slight whitening
6	crazed	orange peel	90	19	21	
7	4/dense	2/med.dense	96	86	81	discolored
9	6/med. dense	6/med dense	93	5.6	16	
10	10	10	61	50	62	discolored

(a) Small cluster at waterline in center only, 6-8/medium dense.

2.4.2 Hot Diesel Oil (Kerosene)

The kerosene immersion test was run in a five gallon container whose top was secured using a large circular clamp fitting over the lip of the bucket. The 4″ x 8″ panels were separated from each other by means of glass rods at the top and bottom of each panel. The container was filled with diesel oil #1 (kerosene) half way up the length of the panels. This assembly was placed in a steam over at 160°F. A steam oven was used for safety reasons. Panels were examined weekly and their condition noted. All of the coated panels performed well in this test and there were no film defects such as softening or blistering noted on any of them. Results are shown in Table IV. The scale in ASTM D-714-56 was used for the rating for blistering. Gloss measurements were made using a 60° Gardner Glossmeter on two panels of each paint on the immersed portion and on the uncovered portion. Results are included in Table IV.

Photograph 2.4.2 illustrates the results on coatings 2, 3 and 5, all coal tar-epoxy types.

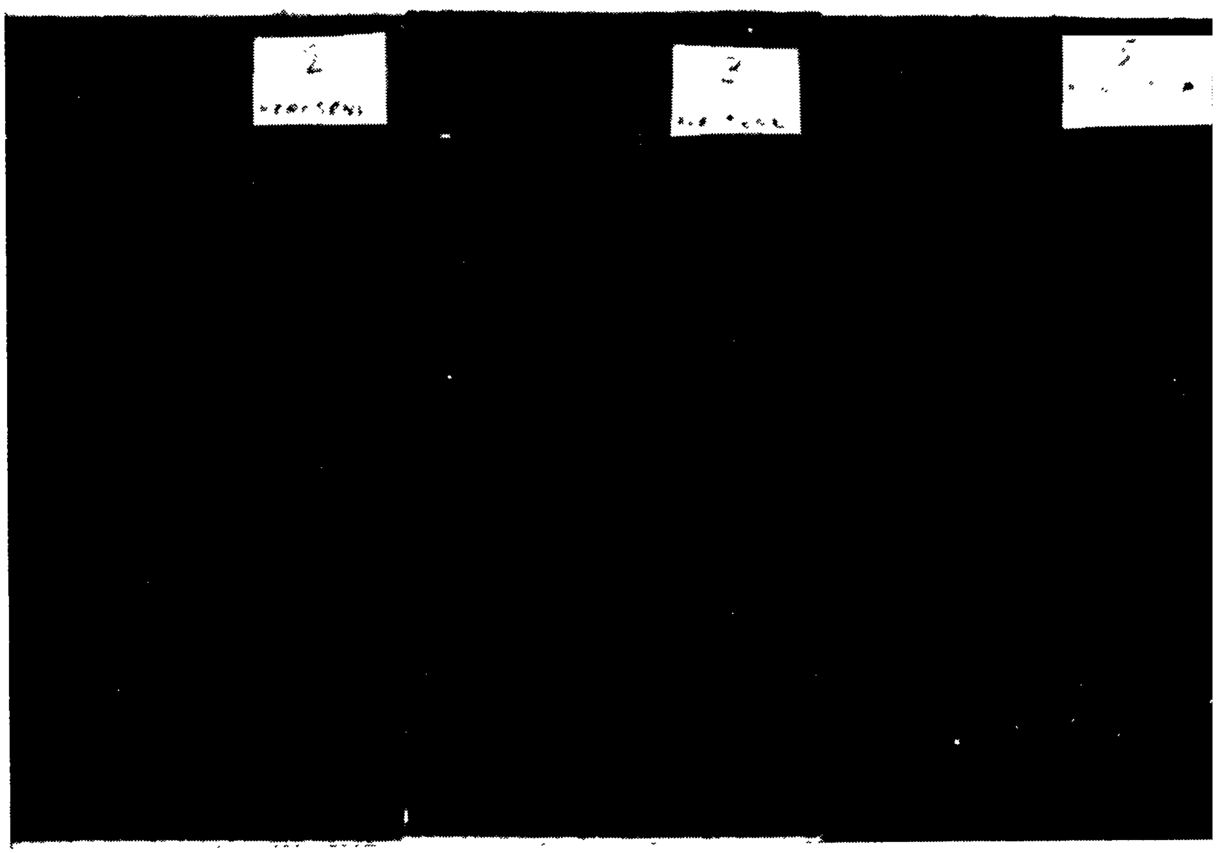


Figure 2.4.2 Hot Diesel Oil Test

TABLE IV

RESULTS OF DIESEL OIL IMMERSION - 500 HOURS

Panel Designation	ASTM D-714-56 Blistering Above	ASTM D-714-56 Blistering Below	% Average Gloss New Panels	% Average Gloss After Immersion Above	% Average Gloss After Immersion Below	Comments
Z	10	10	77	44	37	
0	10	10	matte finish			
1	10	10	44	42	49	
2	10	10	79	46	50	
3	10	10	37	32	26	
4	10	10	matte finish			
5	10	10	86	53	47	
6	10	10	90	31	32	
7	10	10	96	83	80	
9	10	10	93	88	91	
10	10	10	61	51	39	

2.4.3 Salt Spray

The salt spray test was run according to ASTM B-117-73, using 5% NaCl solution and atomizing to spray as prescribed in the test method.

The 4“ x 8“ panels were set into a wooden holder and placed in the salt-spray chamber whose temperature was 92 ± 2°F. Panels were examined weekly and their condition noted. The results are listed in Table V. It must be mentioned that no rusting occurred on any of the coatings other than along the score marks and in some cases near edges where the backing material did not provide adequate protection. Blistering where seen was only along or near score marks. Coating Z (polyurethane type) was probably the least affected in the salt spray test. Of the controls, coating #1 (ketimine-activated epoxy) appeared to be affected the most, possibly because of its color (white) although it did show medium dense blistering near the score marks and under film corrosion to 1/4” under the lines. Coatings #2 and #3 (coal tar-epoxies) stood up well, with very slight under film corrosion along score marks; #3 is the control for this type coating. Of the amine-adduct cured epoxies (#O and #6), the control (#O) showed less blistering at or near the score lines and less under film corrosion at the score marks. Blister ratings used the scale in ASTM D714-56.

Photographs 2.4.3 and 2.4.4 illustrate some of these results - - as a class of coating, the coal tar-epoxy was next to the urethane type, with the amine-adduct epoxy more inclined to blister on scored portions.

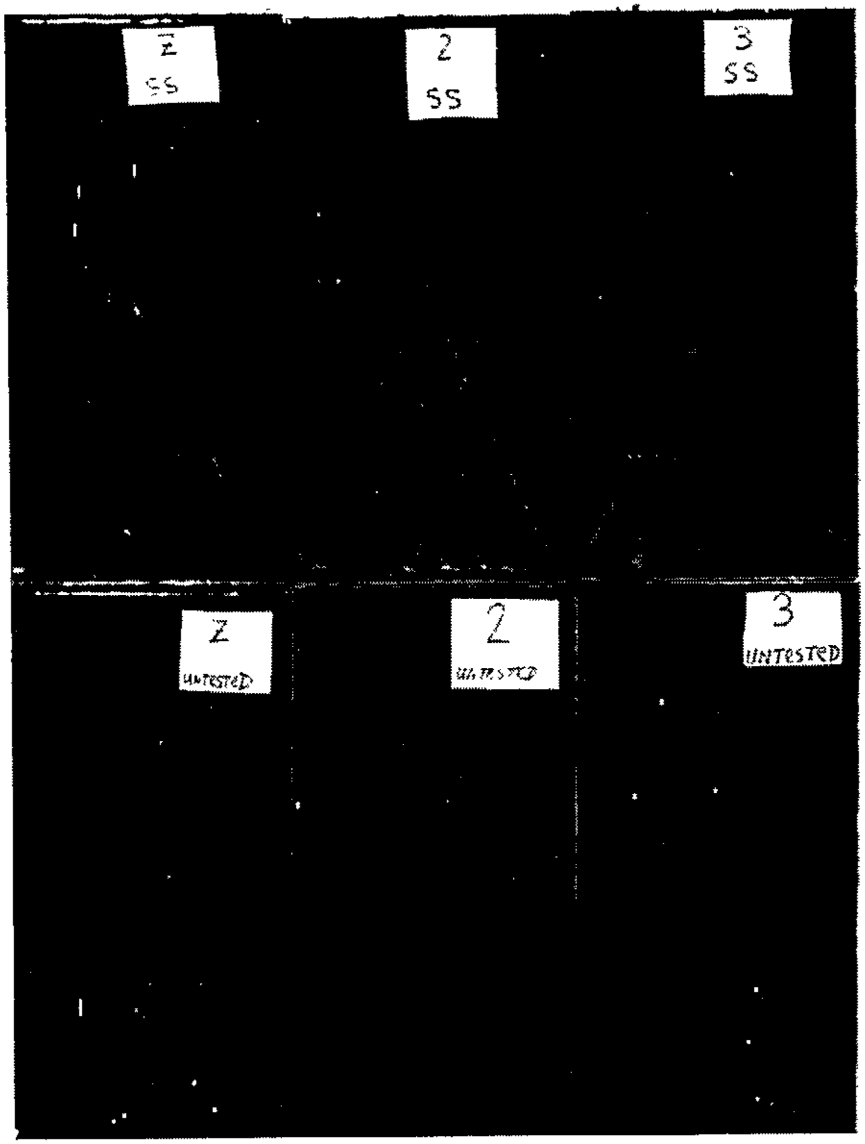


Figure 2.4.3 Salt Spray Test

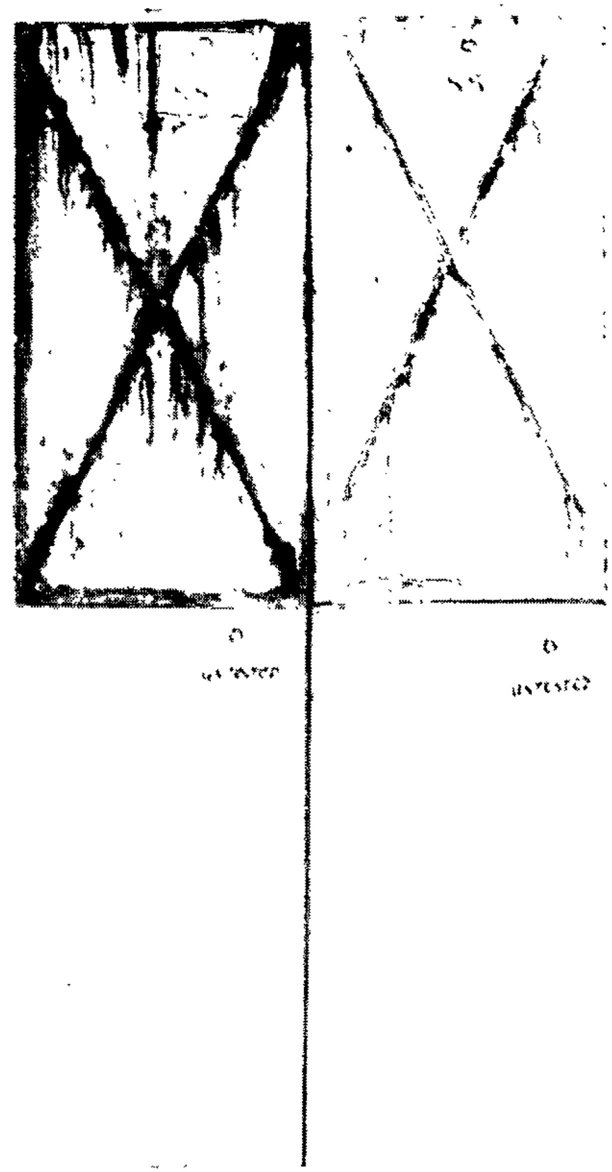


Figure 2.4.4 Salt Spray Test

TABLE V

RESULTS OF SALT SPRAY - 500 HOURS

Panel Designation	Blistering ASTM D714-56	Comments
Z	10	Slight rust on cut lines and edges. No underfilm corrosion at intersection of cut lines.
0	10	Rust on cut lines & edges. Black, tar-like substance on lower cut lines. Underfilm corrosion under tar-like substance, but not found anywhere else.
1	2/med. dense	Blisters only near cut lines, within 1/4". Rust on cut lines and edges. Small cracks on edges of blisters. Underfilm corrosion [a].
2	10	Tar-like substance evident on some places on cut line. Very slight amount of underfilm corrosion, extends 1/8" from intersection point of cut lines.
3	10	Tar-like substance on cut line, Rust on cut lines and edges. Very slight amount of underfilm corrosion, extending less than 1/8" from intersection points.
4	4-6/med. near cut lines 4/few over rest of surface	Tar-like substance on cut lines. Rust on cut lines and edges. Underfilm corrosion extends 3/16" from intersection of cut lines.
5	6/few	Blisters only near cut lines, within 1/4". Rust on cut lines and edges. Underfilm corrosion extends 1/8" from intersection of cut lines.
6	2/few	Blisters only near cut lines, within 1/4". Rust on edges and cut lines. Tar-like substance on cut lines. Underfilm corrosion extends 1/16" from intersection point.
7	2-6/med. dense	Blisters only near cut lines, within 1/4". Tar-like substance on cut lines. Rust on cut lines and edges. Underfilm corrosion extends.[b]
9	2-4/medium	Blisters only near cut lines, within 1/4". Tar-like substance on cut lines. Rust on cut lines and edges. Underfilm corrosion extends.[c]
10	2-6/dense	Blisters only near cut lines, within 1/4". Tar-like substance on cut lines. Rust on cut lines. Underfilm corrosion extends 1/4".[d]

(a) Extends 1/4" in all directions of intersection of the cut lines.
(b) 5/16" due to blister adjacent to intersection point, otherwise it extends only 1/8".
(c) 1/4" from intersection of cut lines.
(d) From intersection of cut lines.

2.4.4 Pressure Immersion

pressure immersion testing was run at 40 psi for six weeks on 4" x 8" panels. The panels were totallY immersed in water and were separated using glassrods on the bottom and top. Panels were examined each week and their condition noted. Results are shown in Table VI.

Although the primary rating for failure was by blistering, some whitening and chalking was shown, particularly on the black coatings. On blistering, all of the coatings appeared to be unaffected by the exposure condition and were so noted in Table VI; all rating 10 on the ASTM D714-56 scale. Photograph 2.4.5 shows the whitening and loss of gloss, particularly evident on control coating #3 (coal tar-epoxy). The other coal tar epoxy coatings #2 and #5 were less affected al though both showed a degree of whitening along the vertical edges. The proper concept from this is that the exposure to water under 40 psi pressure did not produce a single case of loss of protection for the metal substrate.

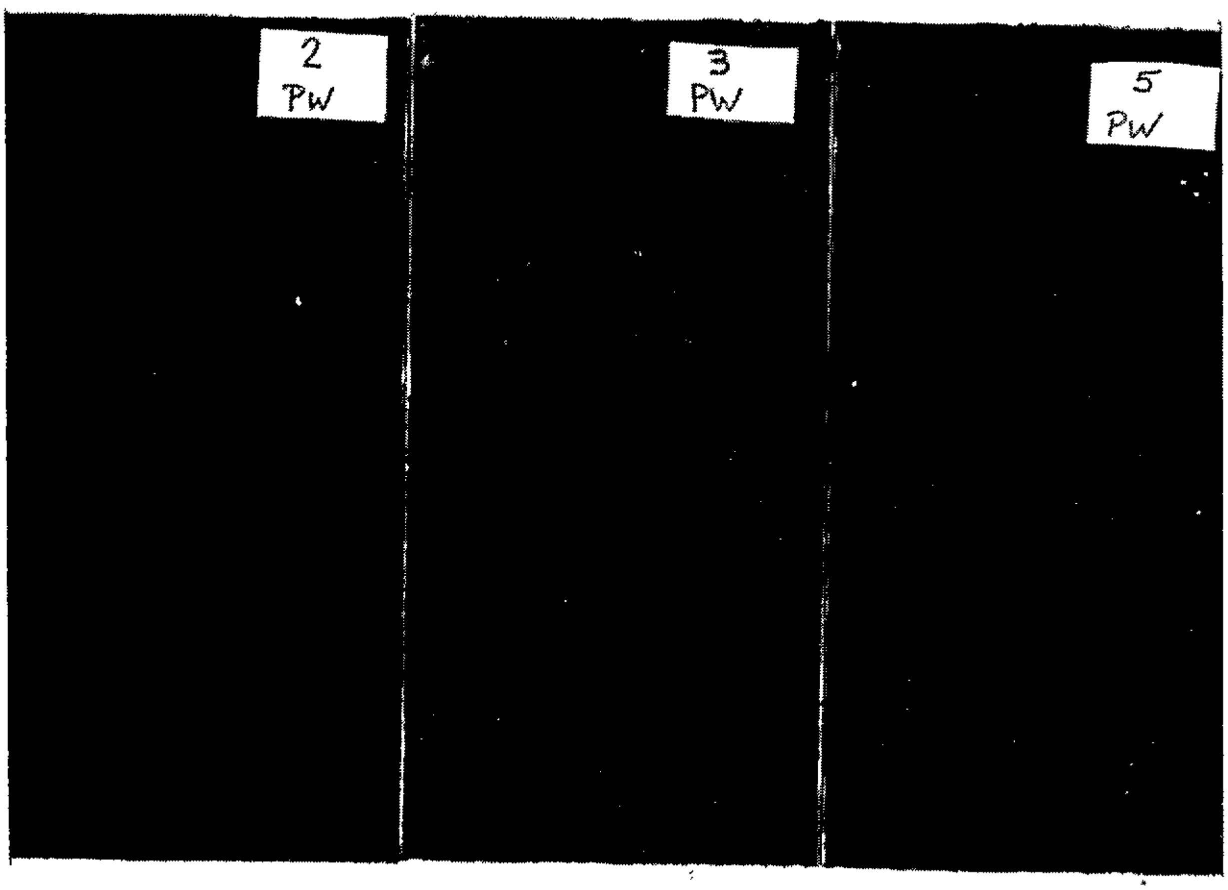


Figure 2.4.5 Pressure Immersion Test

TABLE VI

RESULTS OF PRESSURE TEST

40 psi., 6 weeks

Panel Designation	Blistering ASTM D714-56
Z	10
0	10
1	10
2	10
3	10
4	10
5	10
6	10
7	10
9	10
10	10

2.4.5 Accelerated Weathering

Duplicate 2″ x 6″ panels of each coating were fastened by Nicrome wires to aluminum sheet which was first covered with screening to allow drainage of the water which is sprayed. The panels were placed in an Atlas carbon-arc Weather-Ometer. The test panels are exposed to 102 minutes of carbon-arc light without water and to 18 minutes of light with water spray during each 2-hour cycle. The temperature is kept at 145° ± 9°F. In this manner the coatings are subjected to accelerated weathering. Panels were examined every week and their conditions noted. Results are shown in Table VII. The table details results on this 1000 hours test. Failure mode was by chalking (surface disintegration), some discoloration (on light colored panels), and some pinholes (#6, #7, #10). Only one coating (#4) showed crazing. Coatings 1, 0, Z, 3, 5 and 7 showed only chalking and gloss loss. All except coating Z showed a degree of surface roughness. Protection of the metal surface appeared to be adequate in all cases. In this test, coatings #4, #6, #9 and #10 were rated as poorer than the rest.

Photographs 2.4.6 and 2.4.7 show the condition of each coating tested on a “before” and “after” basis.

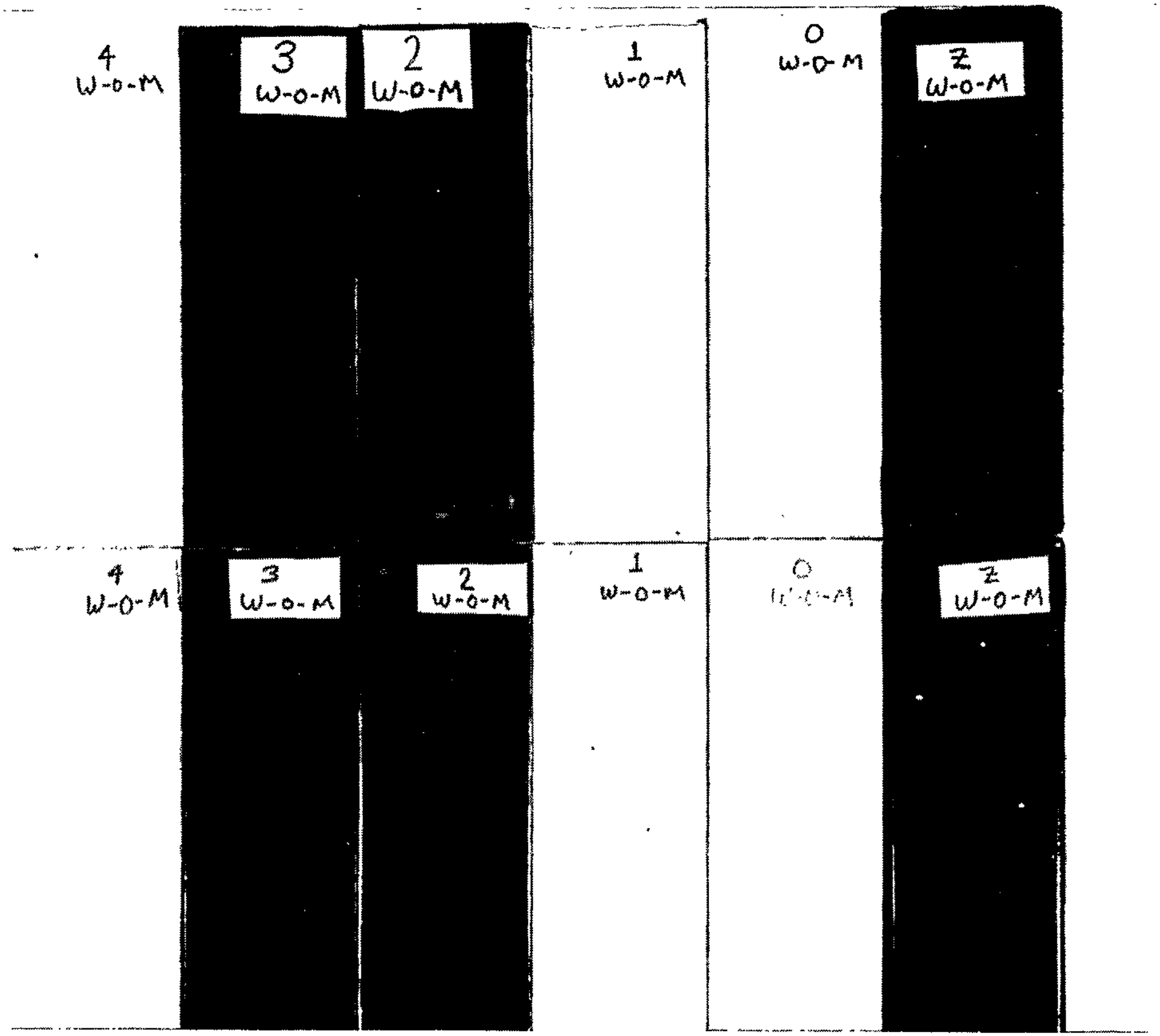


Figure 2.4.6 Accelerated Weathering Test

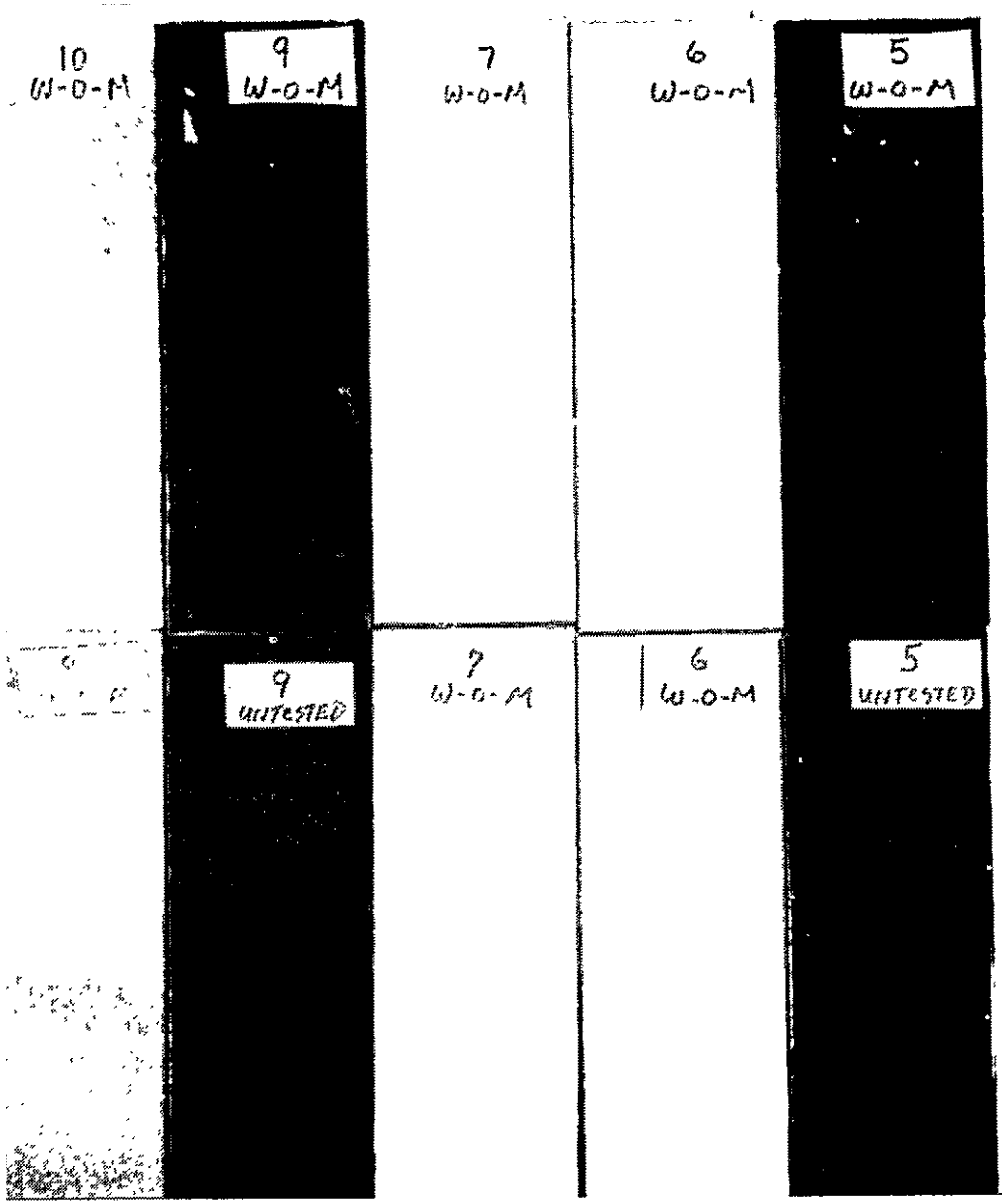


Figure 2.4.7 Accelerated Weathering Test

TABLE VII

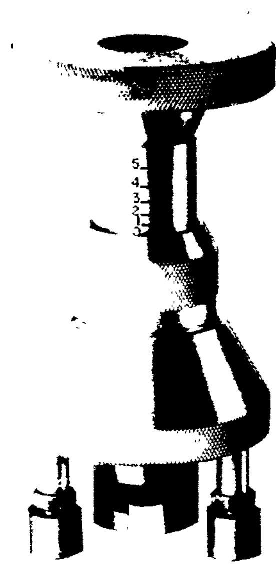
ACCELERATED WEATHERING RESULTS

1000 HOURS

PANEL DESIGNATION	BLISTERING	RUST	OBSERVATIONS
Z	10	10	No blistering or crazing. Chalking and surface roughness. Loss in glass.
0	10	9	No blistering or crazing. Surface degradation, chalking, slightly rough surface. Few tiny rust spots, along with rust on a defect in a small section in the coating. Loss in gloss.
1	10	10	No blistering or crazing. Slight yellowing,less gloss,slightly rough surface. Slight chalking.
2	10	9	No blistering or crazing. Chalking, patches of red, rough surface.
3	10	10	No blistering or crazing. Chalking, loss in gloss, rough surface.
4	10	10	No blistering. Craze lines over entire surface of coating. Chalking with patches of light gray. Rough surface.
5	10	10	No blistering or crazing. Chalking, rough surface, loss in gloss.
6	10	9+	No blistering or crazing. Tiny pinholes in coating are evident. Chalking, rough surface, loss in gloss.
7	10	9+	No blistering or crazing. Slight chalking, loss in gloss, and slightly rough surface.
9	10	9	No blistering or crazing. Pinholes in coating are evident over entire surface. Some pinholes were there before exposure, but not in as many as now visible. Loss in gloss, rough surface and chalking.
10	10	9	No blistering or crazing. Some pinholes, rough surface, chalking and loss in gloss.

2.4.6 Adhesion

Adhesion was measured using an Elcometer Adhesion Tester, purchased from KTA Instruments, Coraopolis, PA., model 106/2 scale range 0-1,000 pounds/sq. in. In this method an aluminum dolly is cemented to the surface of the coating by means of a suitable adhesive such as an epoxy. When the adhesive is cured, the claw of the instrument is placed under the dolly head. The hand wheel on top of the instrument is then tightened until the dolly pulls off and the scale is read. A picture of the tester is shown.



Results of the adhesion tests are shown in Table VIII.

The photographs which follow show some of the test panels from the adhesion testing.

The material which precedes the photograph contains graphic description of the observations in adhesion testing.

Table VIII shows the point and pressure needed to remove paint (or coating) from the test surface. Note that ratings are in absolute terms, based on point of failure and are not in terms relative to any standard pre-stated conditions.

DESCRIPTION OF FAILURES IN ADHESION TEST

Coating #		
Z	500 psi	None of the paint was removed. Most of the epoxy cement was left on the paint.
	900 psi	About 50% of the paint was removed, (loss of adhesion), cement on the rest of the dolly.
	950 psi	About 60% of the paint was removed (loss of adhesion), epoxy cement on the rest of the dolly.
0	400 psi	Dolly surface covered with paint. 90% loss of adhesion.
	350 psi	Dolly surface covered with paint. 90% loss of adhesion.
	375 psi	Dolly surface covered with paint. 90% loss of adhesion.
	275 psi	About 10% of the paint was removed (loss of adhesion), epoxy cement on the rest of the dolly.
1	700 psi	About 60% loss of adhesion, epoxy cement seen both on coating and dolly.
	500 psi	About 50% loss of adhesion, epoxy cement seen both on coating and dolly.
	475 psi	About 40% loss of adhesion, epoxy cement seen both on coating and dolly.
	875 psi	About 40% of the paint was left on the dolly, epoxy cement both on coating and dolly.
2	600 psi	About 50% of paint loss adhesion, epoxy cement on the rest of the dolly.
	400 psi	About 15% of the paint lost adhesion, epoxy cement on th rest of the dolly.
	425 psi	About 15% of paint lost adhesion, epoxy cement on the rest of the dolly.
	270 psi	About 10% of the paint lost adhesion, epoxy cement on the rest of the dolly.

Description of Failures in Adhesion Test - Continued

Coating #		
3	700 psi -	About 75% of paint lost adhesion, epoxy cement on the rest of the dolly.
	675 psi -	About 60% of paint lost adhesion, epoxy cement on the rest of the dolly.
	600 psi -	About 30% of paint lost adhesion, epoxy cement on the rest of the dolly.
	400 psi -	About 10% of paint lost adhesion, epoxy cement on the rest of the dolly.
4 (a)	200 psi -	90% of paint lost adhesion
	300 psi -	90% of paint lost adhesion
	375 psi -	90% of paint lost adhesion
5	1000 psi -	Dolly could not be removed with Elcometer instrument.
	900 psi -	About 15% of paint was left on the dolly, epoxy cement on the rest of the dolly.
6	425 psi -	None of the paint removed. Half of the epoxy cement remained on paint, balance on dolly.
	675 psi -	None of the paint removed. Half of the epoxy cement remained on paint, balance on dolly.
	600 psi -	None of the paint removed. Half of the epoxy cement remained on paint, balance on dolly.
7	500 psi -	No paint removed.
	625 psi -	No paint removed.
	900 psi -	About 40% of paint lost adhesion, epoxy cement on the rest of the dolly.
9	575 psi -	About 80% of paint removed down to base metal.
	550 psi -	About 75% of paint was removed down to base metal.
	500 psi -	About 50% of paint was removed down to base metal.

Description of Failures in Adhesion Test - Continued

Coating #

10 800 psi - About 30% of paint lost adhesion.

575 psi - Epoxy cement on both dolly and paint.

475 psi - Epoxy cement on both dolly and paint, no loss of adhesion.

(a) The epoxy adhesive apparently softened the polyester based paint.

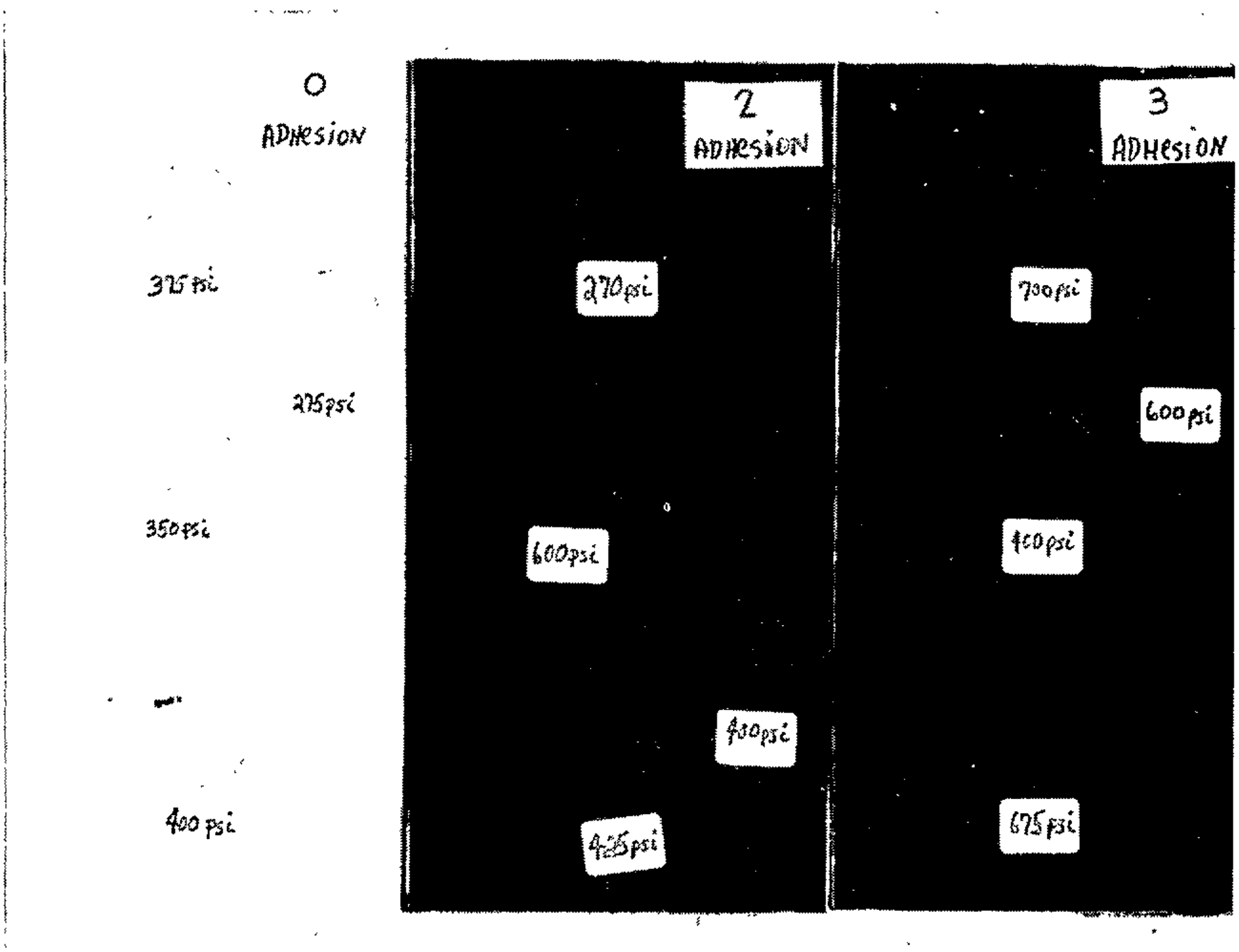


Figure 2.4.8 Adhesion Test

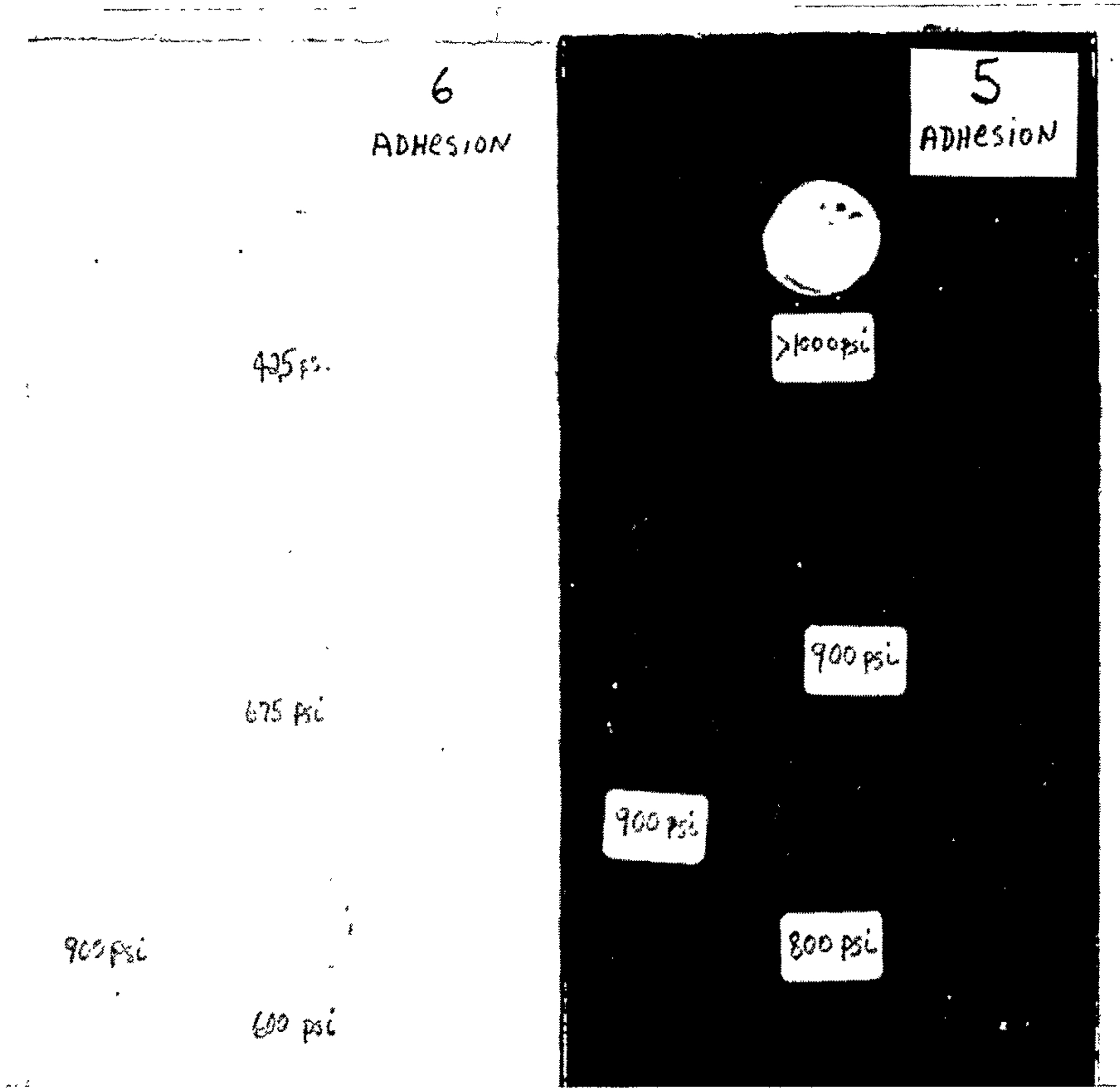


Figure 2.4.9 Adhesion Test

TABLE VIII

ADHESION TEST

Coating Designation	To 50% Adhesion Failure (psi)	Rating(2)	
		Absolute	Relative
Z	950	9.5	(2)
0	400	4.0	(10)
1	875	8.8	(5)
2	600	6.0	(8)
3	700	7.0	(7)
4	375	3.8	(11)
5	>1000(1)	>10	(1)
6	900	9.0	(3)
7	900	9.0	(3)
9	575	5.8	(9)
10	800	8.0	(6)

(1) Dolly could not be removed with Elcometer.
(2) These are absolute ratings. Ratings are obtained by dividing adhesive failure value in psi by 100.